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(54) **Polyamide compositions for
textile fibres**

(57) The present invention relates to
novel compositions formed of a
homopolyamide and of
polyetheramides, the polyetheramides

being obtained by condensing
together polyether blocks and
polyamide blocks and having
molecular weights between 10,000
and 40,000. The compositions are
formed into yarns and fibres for textile
applications.

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SPECIFICATION

Textile fibres and materials displaying hydrophilic properties

The present invention concerns fibers displaying hydrophilic properties and also covers textile materials made up from these fibers.

5 Numerous polymers and copolymers are already known to obtain fibers from which textile materials are manufactured. The drawback of these existing textile materials is however that they are rough to the touch and crease easily. 5

Research in order to obtain textile materials more pleasant to the touch (i.e. a softer touch), and less creasable, has shown that these properties can be particularly obtained from fibres (and thus textile materials which they make up) displaying hydrophilic properties presenting furthermore antistatic properties. 10

In the past it has been noted that 4-nylon fibers or Ultron fibers (commercial name of antistatic 6,6 textile fibers marketed by the company MONSANTO and whose central channel is intermittently clogged by wax) absorb relatively large amounts of water and thus lead to textile materials having hydrophilic properties, thus only slightly creasable and softer to the touch, and having antistatic properties. 15

These materials are not however, satisfactory in so far as, on the one hand, 4-nylon tends to decompose easily in spinning conditions and leads to yellow-tinged textile materials and, on the other hand Ultron is relatively expensive to produce, difficult to dye (due to the wax present therein) and easily loses its antistatic properties, properties which are equally important for textile materials. 20

The present invention concerns fibres suitable for the obtention of a textile material displaying hydrophilic properties, i.e. soft to the touch, similar to that of cotton, and displaying low creasability as well as simultaneously having antistatic properties. These fibers lead to texturable, knittable fibres, to the obtention of textile materials and articles of clothing, easy to dye and having a behaviour similar to that of cellulosic fibres. The textiles materials according to the invention can be boiled, although it is preferable to wash them at temperatures between 30° and 60°C. 25

The present invention concerns textile fibres obtained from a composition formed of a homopolyamide and a polyetheramide, the polyetheramide being formed of the product obtained from condensation in the presence of Ti, Zr or Hf — based catalysts, of polyamide blocks having reactive extremities with polymers blocks having reactive extremities, such as, among others: 30

1 — polyamide blocks having dicarboxylic chain-ends with either polyetherdiol blocks, or polyetherdiamine blocks (by cyanoethylation and hydrogenation of polyetherdiol);

2 — polyamide blocks having diamino chain-ends with polyether blocks having carboxylic chain-ends,

35 the molecular weight of the said polyetheramides being comprised between 10.000 and 40.000. 35

According to one embodiment of the invention, polyetheramide is formed of the product obtained from copolycondensation of dicarboxylic polyamides with polyoxyethylenediol.

According to a further embodiment of the invention, the overall content of the said compositions in polyoxyethylenediol is comprised between 15 and 50%.

40 According to another embodiment of the invention, the homopolyamide results from the polycondensation of lactams or amino-acids and preferably comprises a homopolyamide belonging to the list formed from PA—6, PA—6.6, PA—11, PA—12, PA—6.9, PA—6.10 and PA—6.12. 40

By polyetheramides are meant not only block polyetheramides (i.e. formed of blocks displaying a certain chain length of their various constituents) but also statistic polyetheramides (i.e. formed by

45 random chaining of the various monomeric constituents). 45

The dicarboxylic polyamide is preferably PA—6, PA—6.6, PA—11, PA—12, PA—6.10, PA—6.9 or PA—6.12.

According to a further embodiment of the invention, the polyetheramide is prepared according to the method described in French patents 74—18913 and 77—22678 in the name of the applicant,

50 patents cited by way of reference and whose contents should be combined with the present description 50

The said compositions from which the fibers are obtained preferably contain, among others, 0.3 to 0.8% TiO₂ as well as the standard ingredients used such as antioxidants, anti-ultraviolets, optical azurers, etc.

The present invention concerns, among others, textile application of these compositions and particularly textile materials obtained from the use of fibers, the swelling capacity of which is comprised between 20 to 80%, and more especially between 20 and 40%. 55

Other aims and advantages of the present invention will become evident after reading through the following description and examples, given by way of non-limitative illustration.

EXAMPLE 1

60 In a 1.000 l capacity reactor is charged 150 kg dicarboxylic 6-polyamide having an average molecular weight of 1.500 obtained from polycondensation of ϵ -caprolactam in the presence of adipic acid. 150 kg polyoxyethylenediol having a molecular weight of 1.500 and 0.9 kg tetrabutylorthozirconate are added. The reaction mixture is heated under vacuum of 1 Torr at 150°C for 60

5 hours, with vigorous stirring.

The product (I) obtained displays the following features:

intrinsic viscosity	1.30
melting point	190°C

5 The intrinsic viscosity is determined at 25°C in solution containing 0.5 by weight of metracresol. 5

This product (I) contains 50% by weight polyoxyethylenediol.

In an extruder, 50 parts of product (I) are thereafter mixed with 50 parts of 11-polyamide homopolymers, of textile grade, in the presence of necessary antioxidant additives and anti-ultraviolet.

The component obtained displays the following physical properties:

10	intrinsic viscosity	1.14	10
	melting point	164°C	

The product is thereafter spun with a winding speed of 3.000 m/mn. A thread or yarn is obtained displaying hydrophilic and antistatic properties of which the properties are shown in the table herein-under:

15	thread	165 dtx—30 brins	15
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EXAMPLE 2

In an extruder 30 parts of product (I) are mixed with 70 parts of 11-polyamide homopolymer of textile grade, in the presence of suitable additives.

The product obtained displays the following physical properties:

20	intrinsic viscosity	1.07	20
	melting point	175°C	

The product is spun as in Example 1. It displays the properties shown in Table I hereinunder.

EXAMPLE 3

25 According to operating conditions of Example 1, 150 kg dicarboxylic 12-polyamide (obtained by polycondensation of dodecalactam in the presence of adipic acid), having a molecular weight of 1.500 are caused to react with 150 kg polyoxyethylenediol, having a molecular weight of 1.500, in the presence of 0.9 kg tetrabutylorthozirconate. 25

Product (II) obtained displays the following characteristics:

30	intrinsic viscosity	1.45	30
	melting point	170°C	

This product contains 50% by weight of polyoxyethylenediol initially used.

In an extruder, 50% of product (II) is mixed with 50 parts of 11-polyamide homopolymer, of textile grade, in the presence of appropriate additives.

The product obtained displays the following physical properties:

35	intrinsic viscosity	1.45	35
	melting point	170°C	

The product is spun as in Example 1. It displays the properties shown in the table herein-under.

EXAMPLE 4

40 In an extruder 30 parts of product (II) are mixed with 70 parts of 11-polyamide homopolymer, of textile grade, in the presence of appropriate additives. 40

The product obtained displays the following physical properties:

intrinsic viscosity	1.12
melting point	180°C

The product is spun as in Example 1. It displays the properties shown in table I herein-under.

45 EXAMPLE 5 45

In an extruder 30 parts of product (I) are mixed with 70 parts of 6-polyamide homopolymer, of textile grade, in the presence of suitable additives.

The obtained product displays the following physical properties:

intrinsic viscosity 1.10
melting point 210°C

The product is spun as in Example 1. It displays the properties indicated in table I, herein-under.

TABLE I

Example	Title (dtex)	Absorption rate of H ₂ O (%)	Swelling capacity (%)	Discharge period (s)
6 pure PA. 11 sample	165	1.1	5.9	1860
1	165	4.3	36.4	3
2	165	2.5	23.5	6
3	165	2.0	23.1	3
4	165	1.9	16.3	8
5	165	3.8	32.5	3
7 ULTRON	165	4.5	16.8	39
8 COTTON	165	6.9	43.4	43

5 This table contains the obtained results for the different compounds tested in Examples 1 to 5, as 5
well as the obtained results for samples comprising pure 11-polyamide, Ultron and cotton (Examples 6
to 8). The data determined are the water absorption rate (%), swelling capacity (%) and discharge period
(1), (or time required for the semi-discharge to take place). The discharge period is determined under a
relative humidity rate of 40% and a temperature of 20°C with the use of the static voltmeter device
10 manufactured by Rothschild. The test sample on which this test is carried out is fixed between the jaws 10
of the device; the potential difference between these clamps is 100 volts and the necessary time is
measured for the potential difference to be reduced to 50% its initial power, i.e. 50 volts. The antistatic
character is that proportionally better as the discharge period is lower.
15 In the present description and the claims to which it gives rise is meant by:
swelling capacity: the rate of swelling measured after immersion, during 4 hours in distilled water 15
at 20°C and then centrifugation,
water absorption:

$$\frac{\text{weight under normal atmosphere-anhydrous weight}}{\text{anhydrous weight}} \times 100 \text{ ratio}$$

determined after equilibrium under normal atmosphere (humidity level = 65°C,
20 temperature = 20°C). 20
Of course, the present invention is in no way limited to the embodiments described and
represented; it can be adapted to numerous variations available to the man skilled in the art, according
to the applications foreseen and without departing from the spirit of the invention, as defined in the
accompanying claims.

CLAIMS

1. Textile fibres displaying hydrophilic properties obtained from compositions formed of a homopolyamide and a polyetheramide, the polyetheramide being formed of the product of polycondensation, in the absence of Ti, Zr or Hf, based catalysts, of polyamide blocks having reactive
- 5 extremities with polyether blocks having reactive extremities, such as, among others: 5
- 1 — polyamide blocks having dicarboxylic chain-ends with ether polyetherdiol blocks, or polyetherdiamine blocks (by cyanoethylation and hydrogenation of the polyetherdiol);
- 2 — polyamide block having diamino chain-ends with polyether blocks having carboxylic chain-ends, the molecular weight of the said polyetheramides being comprised between 10.000 and 40.000.
- 10 2. Textile fibres according to claim 1, wherein the polyetheramides are block polyetheramides. 10
3. Textile fibres according to claim 1, wherein the polyetheramides are statistic polyetheramides.
4. Textile fibres according to claim 1, wherein the said compositions are formed of a homopolyamide and a polyetheramide, this polyetheramide being formed of the product of copolycondensation of dicarboxylic polyamides with polyoxyethylendiols.
- 15 5. Textile fibres according to one of claims 1 to 4, wherein the total content of the said compositions in polyoxyethylenediol is comprised between 15 and 50%. 15
6. Textile fibres according to one of claims 1 to 4, wherein the homopolyamide results from the polycondensation of lactams or amino acids.
7. Textile fibres according to claim 6, wherein the homopolyamide belongs to the list formed of
- 20 PA—6, PA—6.6, PA—11, PA—12, PA—6.9, PA—6.10 and PA—6.12. 20
8. Textile fibres according to one of claims 1 to 7, wherein the carboxylic polyamide comprises PA—6, PA—6.6, PA—11, PA—12, PA—6.10, PA—6.9 or PA—6.12.
9. Textile fibres according to one of claims 1 to 8, wherein their swelling capacity is comprised between 20 to 80%.
- 25 10. Textile fibres according to claim 9, wherein the said swelling capacity is comprised between 20 and 40%. 25
11. Textile materials obtained with the use of textile fibres according to one of claims 1 to 8.
12. Textile fibres substantially as hereinbefore described with reference to Example 1 herein.
13. Textile fibres substantially as hereinbefore described with reference to Example 2 herein.
- 30 14. Textile fibres substantially as hereinbefore described with reference to Example 3 herein. 30
15. Textile fibres substantially as hereinbefore described with reference to Example 4 herein.
16. Textile fibres substantially as hereinbefore described with reference to Example 5 herein.
17. Any novel feature or combination of features described herein.